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# EUROPEAN PATENT APPLICATION

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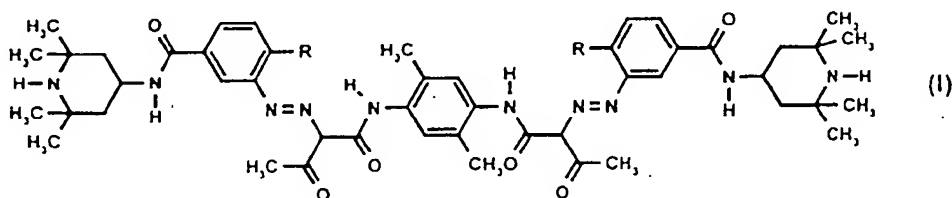
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(54) Pigment for warpage-free polyolefins coloration

(57) Use of a pigment of formula (I)



where both R are Cl or COOCH<sub>3</sub>  
for the coloration of polyolefins which do not warp.

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## Description

[0001] This invention relates to the use of a pigment for the warpage-free coloration of polyolefins.

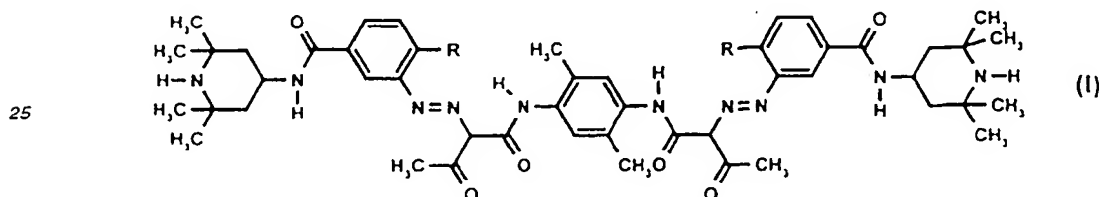
[0002] Unexpected shrinkage and warpage problems can occur when organic pigments are used for the coloration of polyolefins, especially high density polyethylene. This may be due to changes in crystallization behavior due to the presence of pigments. A number of studies have shown that pigments can affect both the size of the crystallites and the rate of crystallization by acting as nucleating agents, inducing crystallization at higher temperatures than expected.

[0003] All these aspects have an important commercial issue. For example the coloration of bottle crates is a very important market for a pigment producer. A pigment has to fulfill an important condition when used in high density polyethylene application: it must not influence the crystallization process of the polymer. Such an influence can cause shrinkage or warpage, showing deformations of the injected article and deterioration of the mechanical properties.

[0004] In order to avoid this problem, when using organic pigment for the coloration of polyolefins, surface treatments have been developed in order to modify the surface of the pigment : precipitation on the surface of the organic particles of a thin layer of a metal oxide (e.g. zirconium, silicium or aluminium) or adsorption or precipitation of some polar polymers (polyacrylates or cellulose derivatives) on the pigment surface. However these treatments are not always efficient.

[0005] It has now been found that light stabilizing azo-pigments of formula (I) do not provoke warpage when used in high density polyethylene.

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where both R are Cl or COOCH<sub>3</sub>.

[0006] The invention, therefore, resides in the use of a pigment of formula (I) for the coloration of polyolefins which do not warp and provides warpage-free polyolefins, more preferably high density polyethylene (HDPE), which have been colored with a pigment of formula (I).

[0007] When coloring polyolefins with a pigment of formula (I) the usual amounts can be used, i.e. from 0.01 to 30% by weight, preferably from 0.1 to 10% by weight based on the material to be colored.

**EXPERIMENTAL PART**

[0008] The testing method is the following:

- Injection mould 10 test plates 60 x 60 mm in HDPE with and without 0.1 % pigment
- processing temperature 220 °C and 280°C
- result: difference of the length and width measurement between test plates in natural HDPE and in HDPE colored with 0.1% pigment.

$$\% \text{ FV} = (\% \text{ vertical shrinkage} - \% \text{ horizontal shrinkage}) / (\% \text{ horizontal shrinkage}) \times 100\%$$

% FV = 0 - 10% : good

% FV = 10-20% : suitable

% FV > 20 % : unsuitable

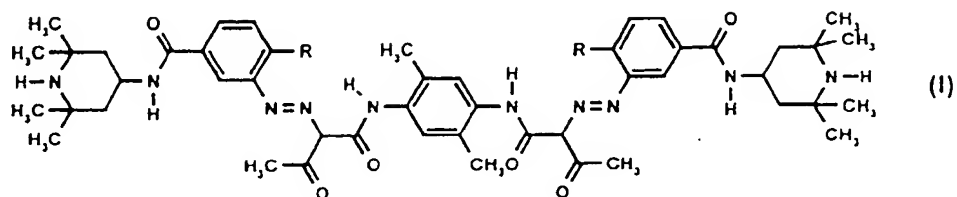
	% FV at 220°C	% FV at 280°C
R = Cl	12.29	12.74
R = COOCH <sub>3</sub>	8.21	1.81

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[0009] With the pigment of formula (I) an excellent result is obtained regarding warpage of the test plates.

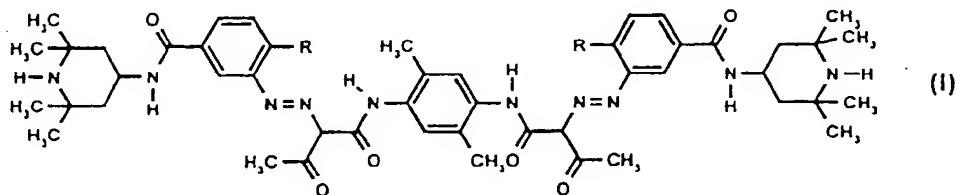
# Claims

1. Use of a pigment of formula (I)



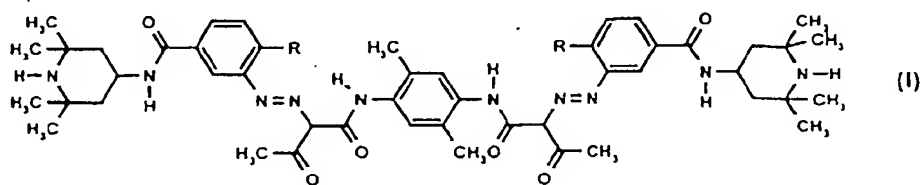
where both R are Cl or COOCH<sub>3</sub>  
for the coloration of polyolefins which do not warp.

2. Warp-free polyolefins which have been colored with a pigment of formula (I)



where both R are Cl or COOCH<sub>3</sub>

3. High density polyethylene (HDPE) which does not warp and has been colored with a pigment of formula (I)



where both R are Cl or COOCH<sub>3</sub>



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# EUROPEAN SEARCH REPORT

Application Number  
EP 00 81 0772

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.7)
Y	US 4 838 943 A (BITTERLI PETER ET AL) 13 June 1989 (1989-06-13) * claim 19; examples 6, A-C *	1-3	C08K5/00 C08K5/23 C08L23/00 C08K5/3435
Y	US 5 472 495 A (SCHROEDER JOERG) 5 December 1995 (1995-12-05) * column 2, line 38-47; claims; examples *	1-3	
			TECHNICAL FIELDS SEARCHED (Int.Cl.7)
			C08K
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 6 November 2000	Examiner Friederich, P
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons &amp; : member of the same patent family, corresponding document</p>			

EPO FORM 1503 03 82 (P4/C01)

**ANNEX TO THE EUROPEAN SEARCH REPORT  
ON EUROPEAN PATENT APPLICATION NO.**

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This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on  
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